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Investigation of the furnace effect in cobalt-carbon high-temperature fixed-point cells

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ABSTRACT

Temperatures with associated uncertainties are to be assigned to high-temperature fixed-points. It is important that those uncertainties are large enough to cover the sources of error but not so overstated as to unnecessarily reduce the usefulness of the fixed-points. Concerns have been raised about unexplained differences between the same fixed-point measured in different furnaces; here we investigate the effect of using three different furnaces on the freezing and melting behaviour of two Co-C eutectic fixed point blackbody cavities. Each furnace had significantly different temperature profiles. The results indicate that the most uniform furnace yielded a higher melting temperature by over 100 mK. Investigation of possible confounding effects: the effect of furnace uniformity on melting curve shape, the micro-structure of the ingot, the temperature drop across the cavity wall, the cavity emissivity and the size-of-source effect show that these factors together are too small to account for the overall observed difference.

1. Introduction

There have been suggestions for a number of years that the radiance temperature of the relatively small fixed-point cells used in research into high-temperature fixed-points (HTFPs) can be significantly different depending on the design of the furnace used in their realisation. This ill-understood effect is known as the Furnace Effect (FE). Two recent publications investigated and quantified this effect at the copper point [1,2]. Realistic values of the maximum temperature difference due to the FE are required as input to the assignment of thermodynamic temperature values with uncertainties to HTFPs [3] for two reasons:

- firstly, as the thermodynamic temperature assignment measurements at different institutes were made using different designs of furnace, it is important to be sure that the true melting temperature lies within the assigned temperature range
- secondly, in future use of these HTFPs, uncertainty components will need to be assigned to their realisation and not assessed by measurement.

Determining a reasonable value to assign for such an uncertainty component is not straightforward. A number of different influence factors can be identified as potentially contributing to the furnace effect; among them the radiation thermometer size-of-source effect [4,5], the cavity emissivity, the temperature drop due to radiance loss from the black-body aperture [6,7], and the so-called structure effect [8]. In addition, it has been shown [9] that poor temperature uniformity can lead to an increased melting range and will cause a lower melting temperature, at least if based on the point-of-inflection of the melting curve. Measurements have been made at NPL of cobalt-carbon (Co-C) HTFP cells (nominally with melting temperature 1324 °C) in order to help evaluate the magnitude of uncertainty arising from the FE. Three different furnaces were used, together with two Co-C high-temperature fixed-point cells; one from NPL and one from NIM [10]. The furnace longitudinal temperature profiles were measured so as to position the fixed-point cells in the optimum position. A radiation thermometer with assessed stability and size-of-source effect was used to measure the difference in point-of-inflection temperatures and liquidus temperatures from the two HTFPs using the different furnaces. The magnitudes of the observed differences are compared to the estimated comparison uncertainties.

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2. Equipment

2.1. Cobalt-carbon high-temperature fixed-points

The Co-C HTFPs of NPL and NIM are of similar design and are described elsewhere [10]. In brief, they consist of a graphite crucible with a black-body re-entrant well. Both Co-C points had a length 45 mm, outer diameter 24 mm and a blackbody aperture diameter of 3 mm. They are both filled with nominally 0.99998 by weight cobalt from different lots by the same supplier. Both Co-C points were of sleeved design (they contain an inner thin-walled liner separating the ingot from the main crucible body) without the incorporation of flexible woven graphite sheet [11]. The use of a layer that has high thermal conductivity separating the thin-walled liner from the crucible body is a later development that reduces the temperature range over which the melting transition is observed. We therefore expect the simple sleeved design used here to show worse performance, in terms of melting range and duration, than the currently accepted best practice design.

2.2. Furnaces

Three different types of furnace were used for these measurements; an indirectly heated carbon-carbon (C/C) composite tube furnace, a directly heated graphite tube furnace and an indirectly heated alumina tube furnace.

2.2.1. Indirectly heated C/C composite tube furnace

The Chino IR-80 [12] uses a C/C fibre composite material as a heater element. The furnace operates in argon and its front window is removed and replaced by a N2 purge unit for measurements, so no window correction is required. The furnace has a maximum working temperature of 2500 °C. It is designed specifically for use with HTFP cells. The working tube inner diameter is 27 mm compared to 24 mm fixed-point cell diameter. The fixed-point cell is wrapped with thin graphite felt, wound and tied with graphite string to give a good fit. A series of graphite felt spacers and C/C discs with increasing aperture diameters are placed either side of the fixed-point cell as insulation.

2.2.2. Directly heated graphite tube furnace

The ThermoGauge HT9500 [13] is a fast response directly heated graphite tube furnace. A 25.4 mm inner diameter graphite tube is clamped between two electrodes and is used as both a work tube and the heater element. The tube is shaped so as to increase the heating at the ends and reduce temperature gradients in the middle. The tube is surrounded by graphite-felt insulation and a layer of graphite foil, all in a fused-silica tube. The whole assembly is enclosed by a water-cooled aluminium reflector. An argon gas purge of this assembly means it can operate without a window up to 3000 °C. Its primary use is as a fast to heat and cool calibration black-body source. The working tube inner diameter is 25.4 mm, and the fixed-point cells are wrapped in 0.4 mm thick compressible graphite sheet to bring to the required diameter. Again, a series of graphite felt spacers and C/C discs with increasing aperture diameters are placed either side of the fixed-point cell as insulation.

2.2.3. Indirectly heated alumina tube furnace

The Lenton furnace (so-called after its manufacturer) [14] is a 3-zone open ended alumina tube furnace indirectly heated by silicon carbide heaters. The end zones have two heater elements each, the middle zone has three. The furnace can operate in air at up to 1600 °C, but an argon purge of the work-tube is used to protect the graphite crucible of the Co-C fixed-point cells. It was found possible to leave these in the furnace at high temperatures, up to 1350 °C, continuously for several days without damage to the crucible. The work tube is 25 mm and as with the Thermogauge, 0.4 mm thick compressible graphite sheet is used as packing. Solid, graphite blocks are placed either side of the fixed-point cell, with the end facing the blackbody aperture having a cylindrical concentric viewing hole. Ceramic wool is added behind. Bored out ceramic bricks are placed in front.

In all cases the measurement procedure was the same: once stabilised in the solid (liquid) state, at nominally 20 °C below (above) the eutectic temperature, the furnace set point was ramped at 20 °C per minute to 20 °C above (below) in order to initiate melting (freezing).

2.3. Radiation thermometer

The IKE LP3 radiation thermometer used has been described in detail elsewhere [15]. It is a silicon photodiode detector based instrument with a 1 mm measuring spot at 830 mm distance. It has an apochromatic objective lens that gives low size-of-source effect contribution to the signal. The wavelength of operation was 650 nm with a bandwidth of 14 nm. This pyrometer is calibrated using a copper point and wavelength calibration, and is used to realise the ITS-90 above the silver point at NPL.

3. Known contributing factors

Before coming to the measurements, we first outline other possible confounding factors which have been suggested as the cause or contributing to the furnace effect.

The following are known to have some effect either on the measurement or on HTFP temperature itself: the uniformity of the furnace, the size-of-source effect of the radiation thermometer, the micro-structure of the eutectic alloy and the definition of melting temperature used. These factors have to be considered and combined with the measurement uncertainty in order to determine if a separate unknown cause for the furnace effect needs to be postulated.

3.1. Uniformity of furnace temperature

The furnace temperature profile (FTP) of each furnace were measured. In [9] it was found that positioning a fixed-point cell in a position of poor uniformity could increase the melting range and cause the point-of-inflection value of the melting curve to be lowered. Therefore, the optimum position to install a fixed-point cell in a furnace was taken to be where there was the minimum temperature gradient over the length of the HTFP cell. To find this, a 50 mm long graphite block with a 3 mm hole drilled along its centre was installed in place of the crucible and a type-R or type-B thermocouple was used to measure the temperature profile by moving the thermocouple within the dummy crucible. The amount of insulation and the position in the furnace were investigated to determine the optimum amount of insulation required and position. The Lenton could, in addition, be tuned for best uniformity by adjusting the offsets of the front and rear zones relative to the middle zone.

3.2. Size-of-source effect

Fig. 1 shows the LP3 size-of-source effect (SSE) response, measured by the indirect method [4]. This can be combined with the radiance profile of the source to estimate the contribution to the signal that originates from outside the radiating cavity of the fixed-point cell. As the exact details of the horizontal temperature